IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:	Docket No.: 07620004AA
Darren ROGERS	
Serial No.: 09/733,602	Group Art Unit: 1714
Confirmation No.: 2099	
Filed: December 8, 2000	Examiner: Margaret B. MEDLEY

For: DESIGNED CELLULAR COAL

Mail Stop Petition
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF AUZVILLE JACKSON, JR.

I, Auzville Jackson, Jr., hereby declare as follows:

1. I was the attorney of record from December 8, 2000 the filing date of the above application until September 25, 2002.

2. On December 8, 2000, a specification (22 pages), drawings (5 pages), an executed declaration (2 pages), an executed assignment (2 pages), an assignment recordation coversheet, a transmittal form PTO/SB/05, and a fee transmittal form PTO/SB/17 were filed by Express Mail (Express Mail Label: EK828076037).

3. A copy of the December 8, 2000 filing, transmittal page and Official Filing Receipt are attached as Exhibit A.

4. I did not receive the Notice of Informal Application dated May 16, 2001. A copy is attached as Exhibit B.

5. The failure to reply to the Notice of Informal Application dated May 16, 2001 was unavoidable since I did not receive the Notice.

6. I have reviewed my remaining file to confirm no such Notice was ever received. Also, I am informed by the Attorney now handling the application that his files do not have such a Notice.

7. The deficiencies identified in the Notice of Informal Application of an oath or declaration not identifying the residence of each inventor are not accurate.

8. A copy of the executed declaration and a copy of the assignment identifying each inventor and the city and state of residence accompanied this application filed in the USPTO on December 8, 2000. See Exhibit A.

9. I personally signed the transmittal sheet and mailed the executed declaration and assignment and other papers filed in the original application to the USPTO on December 8, 2000.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code.

Auzville Jackson, Jr.
Reg. No. 17,306

12/23/04
Date

Express Mail Label: EK828076037 US Date of Deposit: 12/8/00

"I hereby certify that this correspondence is being deposited with the United States Postal Service using "Express Mail Post Office to Addressee" service under C.F.R. Section 1.10 on the date indicated below and is addressed to the addressee herein.

Auzville Jackson, Jr. By: Auzville Jackson Jr. Date: 12/8/00

Exhibit A

Please type a plus sign (+) inside this box →

PTO/SB/05 (08-00)
Approved for use through 10/31/2002. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	1391 (TOUCHSTONE)
First Inventor	ROGERS
Title	DESIGNED CELLULAR COAL
Express Mail Label No.	EK828076037 US

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. [X] Fee Transmittal Form (e.g., PTO/SB/17) *(Submit an original and a duplicate for fee processing)*
2. [X] Applicant claims small entity status. See 37 CFR 1.27.
3. [X] Specification [Total Pages 28] *(preferred arrangement set forth below)*
   - Descriptive title of the invention
   - Cross Reference to Related Applications
   - Statement Regarding Fed sponsored R & D
   - Reference to sequence listing, a table, or a computer program listing appendix
   - Background of the Invention
   - Brief Summary of the Invention
   - Brief Description of the Drawings *(if filed)*
   - Detailed Description
   - Claim(s)
   - Abstract of the Disclosure
4. [X] Drawing(s) *(35 U.S.C. 113)* [Total Sheets 5]
5. Oath or Declaration [Total Pages 2]
   a. [X] Newly executed (original or copy)
   b. [ ] Copy from a prior application (37 CFR 1.63 (d)) *(for continuation/divisional with Box 17 completed)*
      i. [ ] DELETION OF INVENTOR(S) Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).
6. [ ] Application Data Sheet. See 37 CFR 1.76
7. [ ] CD-ROM or CD-R in duplicate, large table or Computer Program *(Appendix)*
8. Nucleotide and/or Amino Acid Sequence Submission *(if applicable, all necessary)*
   a. [ ] Computer Readable Form (CRF)
   b. Specification Sequence Listing on:
      i. [ ] CD-ROM or CD-R (2 copies); or
      ii. [ ] paper
   c. [ ] Statements verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

9. [X] Assignment Papers (cover sheet & document(s))
10. [ ] 37 CFR 3.73(b) Statement *(when there is an assignee)* [X] Power of Attorney
11. [ ] English Translation Document *(if applicable)*
12. [ ] Information Disclosure Statement (IDS)/PTO-1449 [ ] Copies of IDS Citations
13. [ ] Preliminary Amendment
14. [X] Return Receipt Postcard (MPEP 503) *(Should be specifically itemized)*
15. [ ] Certified Copy of Priority Document(s) *(if foreign priority is claimed)*
16. [ ] Other: .....

17. If a CONTINUING APPLICATION, *check appropriate box, and supply the requisite information below and in a preliminary amendment, or in an Application Data Sheet under 37 CFR 1.76:*

[ ] Continuation [ ] Divisional [ ] Continuation-in-part (CIP) of prior application No.: _____/_____

Prior application information: Examiner _____ Group / Art Unit: _____

For CONTINUATION OR DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 5b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

18. CORRESPONDENCE ADDRESS

[ ] Customer Number or Bar Code Label (Insert Customer No. or Attach bar code label here) or [ ] Correspondence address below

Name	AUZVILLE JACKSON, JR.				
Address	8652 Rio Grande Road				
City	Richmond	State	VA	Zip Code	23229
Country	USA	Telephone	804/740-6828	Fax	804/740-1881

Name (Print/Type)	AUZVILLE JACKSON, JR.	Registration No. (Attorney/Agent)	17,306
Signature	Auzville Jackson Jr.	Date	12/8/00

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Box Patent Application, Washington, DC 20231.

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FEE TRANSMITTAL for FY 2000

Patent fees are subject to annual revision.

TOTAL AMOUNT OF PAYMENT ($) 395

Complete if Known	
Application Number	
Filing Date	12/8/00
First Named Inventor	ROGERS
Examiner Name	
Group Art Unit	
Attorney Docket No.	1391

METHOD OF PAYMENT (check one)

1. ☐ The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:
   - Deposit Account Number
   - Deposit Account Name
   - ☐ Charge Any Additional Fee Required Under 37 CFR 1.16 and 1.17
   - ☐ Applicant claims small entity status. See 37 CFR 1.27
2. ☒ Payment Enclosed:
   - ☒ Check ☐ Credit card ☐ Money Order ☐ Other

FEE CALCULATION

1. BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
101	690	201	345	Utility filing fee	355
106	310	206	155	Design filing fee	
107	480	207	240	Plant filing fee	
108	690	208	345	Reissue filing fee	
114	150	214	75	Provisional filing fee	

SUBTOTAL (1) ($) 355

2. EXTRA CLAIM FEES

	Extra Claims		Fee from below	Fee Paid
Total Claims		-20** =		x =
Independent Claims		- 3** =		x =
Multiple Dependent				=

**or number previously paid, if greater; For Reissues, see below

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
103	18	203	9	Claims in excess of 20
102	78	202	39	Independent claims in excess of 3
104	260	204	130	Multiple dependent claim, if not paid
109	78	209	39	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($)

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
105	130	205	65	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for *ex parte* reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	380	216	190	Extension for reply within second month	
117	870	217	435	Extension for reply within third month	
118	1,360	218	680	Extension for reply within fourth month	
128	1,850	228	925	Extension for reply within fifth month	
119	300	219	150	Notice of Appeal	
120	300	220	150	Filing a brief in support of an appeal	
121	260	221	130	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,210	241	605	Petition to revive - unintentional	
142	1,210	242	605	Utility issue fee (or reissue)	
143	430	243	215	Design issue fee	
144	580	244	290	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Petitions related to provisional applications	
126	240	126	240	Submission of Information Disclosure Stmt	
581	40	581	40	Recording each patent assignment per property (times number of properties)	40
146	690	246	345	Filing a submission after final rejection (37 CFR § 1.129(a))	
149	690	249	345	For each additional invention to be examined (37 CFR § 1.129(b))	
179	690	279	345	Request for Continued Examination (RCE)	
169	900	169	900	Request for expedited examination of a design application	

Other fee (specify) _____

* Reduced by Basic Filing Fee Paid

SUBTOTAL (3) ($) 40

SUBMITTED BY

Name (Print/Type)	AUZVILLE JACKSON, JR.	Registration No. (Attorney/Agent)	17306	Telephone	804/740-6828
Signature				Date	12/08/00

WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

FORM PTO-1595
1-31-92

RECORDATION FORM COVER SHEET

U.S. DEPARTMENT OF COMMERCE
Patent and Trademark Office

PATENTS ONLY

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To the Honorable Commissioner of Patents and Trademarks: Please record the attached original documents or copy thereof.

1. Name of conveying party(ies):
Darren Rogers and Janusz W. Plucinski

Additional name(s) of conveying party(ies) attached ☐ Yes ☒ No

2. Name and address of receiving party(ies):
Touchstone Research Laboratory, Ltd.
Internal Address: _____

Street Address: RD #1, Box 100B, The Millenium Centre

City: Triadelphia State: WV Zip: 26059

Additional name(s) and address(es) attached ☐ Yes ☒ No

3. Nature of conveyance:
☒ Assignment ☐ Merger
☐ Security Agreement ☐ Change of Name
☐ Other _____

Execution Date: _____

4. Application number(s) or patent number(s):
If this document is being filed together with a new application, the execution date of the application is:

A. Patent Application No.(s)

B. Patent No.(s)

Additional numbers attached ☐ Yes ☒ No

5. Name and address of party to whom correspondence concerning document should be mailed:
Name: AUZVILLE JACKSON, JR.
Internal Address: _____

Street Address: 8652 Rio Grande Road

City: Richmond State: VA Zip: 23229

6. Total number of applications and patents involved: 1

7. Total fee (37 CFR 3.41)..... $ 40
☒ Enclosed
☐ Authorized to be charged to deposit account

8. Deposit account number: _____
(Attach duplicate copy of this page if paying by deposit account)

DO NOT USE THIS SPACE

9. Statement and signature.
To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

AUZVILLE JACKSON, JR.
Name of Person Signing

Auzville Jackson Jr.
Signature

12/8/00
Date

Total number of pages comprising cover sheet: 1

Docket Number 1391(Touchstone)

(Worldwide Rights)

ASSIGNMENT

FULL NAME(S) AND
POST OFFICE ADDRESS(ES)
(including Country)
OF INVENTOR(S)

WHEREAS:
Darren Rogers
4 America Avenue
Wheeling, WV 26003
USA

And

Janusz Wladyslaw Plucinski
416 6th Street
Glen Dale, WV 26038
USA
(hereinafter referred to as ASSIGNOR(S)) have invented certain new and useful improvements in an invention

TITLE OF INVENTION

Designed Cellular Coal

For which an application for United States Letters Patent is being executed and filed concurrently herewith;

WHEREAS:

FULL NAME AND ADDRESS
(including Country) OF ASSIGNEE
(if a Corporation show state and
identify state/Country of Corporation)

Touchstone Research Laboratory, Ltd.
RD #1, Box 100B, The Millenium Centre
Triadelphia, West Virginia 26059

(Hereinafter referred to ASSIGNEE), is desirous of acquiring the entire interest in, to and under said invention and in, to and under Letters Patent or similar legal protection to be obtained therefore in the United States and in any and all foreign countries.

NOW, THEREFORE, TO ALL WHOM IT MAY CONCERN: Be it known that in consideration of the payment by ASSIGNEE to each of said ASSIGNOR(S) of the sum of One Hundred Dollars ($100.00) the receipt and sufficiency of which

is hereby acknowledged, ASSIGNOR(S) hereby sells, assigns and transfers to ASSIGNEE, its lawful successors and assigns, the full and exclusive right, title and interest to said invention in the United States and its territorial possessions and in all foreign countries and to all Letters Patent or similar legal protection in the United States and its territorial possessions and in any and all foreign countries to be obtained for said invention by said application or any continuation, division, continuation-in-part, reexamination, renewal, substitute, extension or reissue thereof or any legal equivalent thereof in a foreign country for the full terms or terms of which the same may be granted; and authorize and request the Commission of Patents and Trademarks of the United States and any official of any foreign country whose duty it is to issue patents or legal equivalents thereto, to issue same for this invention to ASSIGNEE, its lawful successors and assigns.

ASSIGNOR(S) hereby covenant that no assignment, sale, agreement or encumbrance has been or will be made or entered into which would conflict with this assignment and sale;

ASSIGNOR(S) further covenant that ASSIGNEE will, upon its request, be provided promptly with all pertinent facts and documents relating to said application, said invention and said Letters Patent and legal equivalents in foreign countries as may be known and accessible to ASSIGNOR(S) and will testify as tot he same in any interference or litigation related thereto and will promptly execute and deliver to ASSIGNEE or its legal representative any and all papers, instruments, or affidavits required to apply for, obtain, maintain, issue and enforce said application, said invention and said Letters Patent and said equivalents thereof in any foreign country which may be necessary or desirable to carry out the purposes thereof.

IN WITNESS WHEREOF, I/We have hereunto set hand and signed on the date indicated.

SIGNATURE(S)

The signature(s) must correspond with the name(s) of the inventor(s) above.

INVENTOR(S) AND ASSIGNOR(S):	DATE SIGNED	WITNESS(ES)
Darren Rogers	4 DEC 2000	
Janusz Wladyslaw Plucinski	12/4/00	

DOCKET NO. 1391(Touchstone) PATENT

UNITED STATES
COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that: My residence, post office address and citizenship are as stated below next to my name, I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: **Designed Cellular Coal,** the application of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose to the Office all information known to me which is material to the examination of this application as defined in §1.56. I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate having a filing date before that of the application(s) of which priority is claimed.

Prior Foreign Application(s)			Prior Claim	
(Number)	(Country)	(Day/Month/Year Filed)	[ ] Yes	[ ] No
(Number)	(Country)	(Day/Month/Year Filed)	[ ] Yes	[ ] No

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application listed below and insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112. I acknowledge the duty to disclose to the Office all information known to me which is material to the examination of this application as defined in §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

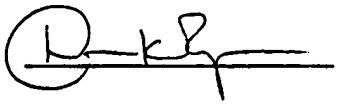
(Application Serial No)	(Filing Date)	(Status: patented, pending, abandoned)
(Application Serial No)	(Filing Date)	(Status: patented, pending, abandoned)

As a named inventor, I hereby appoint Auzville Jackson, Jr., Reg. No. 17,306, as my attorney to prosecute this application, and transact all business in the Patent and Trademark Office connected therewith and to act on my behalf before the competent International Authorities in connection with any and all international applications filed by me and of which I am the sole applicant and to receive payments on my behalf.

Send correspondence to: Auzville Jackson, Jr.
8652 Rio Grande Rd.
Richmond, VA 23229
(804)740-6828 / FAX (804) 740-1881

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of first inventor: **Darren Rogers**

First Inventor's signature: 

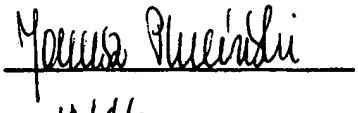
Date: 4 DECEMBER 2000

Address of First Inventor: **4 America Avenue**
Wheeling, WV 26003

Citizenship of First Inventor: **United States of America**

Residence of First Inventor: **4 America Avenue**
Wheeling, WV 26003

Full name of second inventor: **Janusz Wladyslaw Plucinski**

Second Inventor's signature: 

Date: 12/4/00

Address of Second Inventor: **416 6th Street**
Glen Dale, WV 26038

Citizenship of Second Inventor: **Poland**

Residence of Second Inventor: **416 6th Street**
Glen Dale, WV 26038

Designed Cellular Coal

Field of the Invention

The present invention relates to cellular coal products formed from coal and to their methods of production and more particularly to such products that are custom manufactured to provide regions and volumes within their structures that exhibit varying strength, density and or porosity characteristics.

Background of the Invention

U.S. Patent Application Serial No. 09/453,729, filed December 2,1999 entitled "Cellular Coal Products and Processes" describes coal-based cellular or porous products having a density of preferably between about 0.1g/cm^3 and about 0.8g/cm^3 that are produced by the controlled heating of coal particulate preferably up to 1mm in diameter in a "mold" and under a non-oxidizing atmosphere. The porous products produced by these processes, preferably as a net shape or near net shape, can be machined, adhered and otherwise fabricated to produce a wide variety of low cost, low density products, or used in its preformed shape as a filter, heat or electrical insulator etc. Such cellular products have been shown to exhibit compressive strengths of up to about 4000 psi. Impregnation with appropriate materials or the incorporation of various strength improving additives can further increase the compressive, tensile and other properties of these cellular materials.

Further treatment by graphitization yields cellular products that can be used as electrical or heat conductors.

The coal-based products described in the foregoing U.S. Patent Application have many potential uses that are supported by their low densities, high strengths, thermal and electrical conductivities and fluid permeabilities, however, many such applications or uses require combinations of such properties in order to provide the desired product. For example, while low density and relatively high strength may be required for a specified use, attachment of the coal-based foam product demonstrating such low density and relatively high strength may require the inclusion of a very high strength region in a portion of the product part to permit the passage of a fastener that will induce areas of high mechanical stress in the area of fastener passage. Similarly, while fluid permeation of the coal-based cellular product may be desired in one direction, it may be necessary to limit or inhibit such fluid permeation in orthogonal directions to achieve directed fluid flow as through a "pipe". It can also be envisioned that a low-density coal-based product part may require the inclusion of high strength regions to provide structural properties in regions thereof that are subjected to elevated directional mechanical stresses that the bulk of the low density material cannot adequately support.

Thus, it would be highly desirable to be able to custom design various regions of the coal-based cellular product to support various different strength, thermal and electrical conductivity and fluid permeation properties in the various regions of the

coal-based cellular product. Stated differently, it would be highly desirable to possess the ability to provide coal-base cellular products and parts produced from a single cell forming process that exhibit integral: 1) stiffeners or load paths; 2) directed heat or electrical transfer paths; and 3) directed mass transfer paths.

Object of the Invention

It is therefore an object of the present invention to provide a method for the production of coal-based cellular products and parts that exhibit integral, custom designed and varying strength, thermal and electrical conductivity and fluid permeabilities throughout their overall volumes.

Summary of the Invention

According to the present invention, coal-based cellular products having integral stiffeners or load paths, directed heat transfer paths and directed mass transfer paths are provided through the placement of coal-based cells of a different size and/or density than those making up the matrix of the product during manufacture. There is also provided a method for the production of coal-based cellular products possessing these characteristics. The method of the present invention utilizes the ability to select and design such properties through the proper selection and control of cell size and density. Such control of cell size and density is in turn achieved through appropriate selection of starting materials, starting material particle size, mold packing and processing parameters.

Description of the Drawings

Figure 1 is a photomicrograph of a horizontally graded coal-base cellular product.

Figure 2 is a photomicrograph of a vertically graded coal-based cellular product.

Figure 3 is a photomicrograph of a mixed precursor coal-based cellular product.

Figure 4 is a photomicrograph of a radially graded coal-based cellular product.

Figure 5 is a photographic mage of a coal-base cellular product incorporating differing cell sizes in the shape of the letter T.

Figure 6 is a photographic image of a coal-based cellular product incorporating differing cell sizes in the shape of the letter T.

Detailed Description

According to the method described in aforementioned U.S. Patent Application Serial No. 09/453,729, a low density, i.e., from about 0.1 to about 1.0g/cm^3, and preferably from about 0.2 to about 0.5g/cm^3, coal-based cellular product is produced from powdered coal particulate preferably less than about 1mm in diameter by the controlled heating of the powdered coal in a "mold" under a non-oxidizing atmosphere. The starting material coal may include bitumen, anthracite, or even lignite, or blends of these, but is preferably bituminous, agglomerating coals that have been comminuted to an appropriate particle size, preferably to a fine powder below about –60 to –80 mesh. As used herein, the term "coal-based" is meant define that the cellular products described herein are prepared or manufactured by the "controlled swelling" of ground or comminuted coal.

These cellular coal-based products are semi-crystalline or more accurately turbostratically-ordered and largely isotropic i.e., demonstrating physical properties that are approximately equal in all directions, and typically exhibit pore sizes on the order of less than 100μm, although pore sizes of up to 2000μm are possible within the operating parameters of the process described. The thermal conductivities of the cellular coal-based products are generally less than about 1.0 W/m/°K. Typically, the cellular coal-based products of the present invention demonstrate compressive strengths on the order of from about 2000 to about 4000 psi at densities of from about 0.4 to about 0.5g/cm^3.

The production method previously described comprises: 1) heating a coal particulate of preferably small i.e., less than about 200μm particle size in a "mold" and under a non-oxidizing atmosphere at a heat up rate of from about 1 to about 20°C per minute to a temperature of between about 300 and about 700°C; 2) soaking at a temperature of between about 300 and 700°C for from about 10 minutes up to about 12 hours to form a preform or finished product; and 3) controllably cooling the preform or finished product to a temperature below about 100°C. The non-oxidizing atmosphere may be provided by the introduction of inert or non-oxidizing gas into the "mold" at a pressure of from about 0 psi, i.e., free flowing gas, up to about 500 psi. The inert gas used may be any of the commonly used inert or non-oxidizing gases such as nitrogen, helium, argon, CO_2, etc.

It is generally not desirable that the reaction chamber be vented during the heating and soaking operation. The pressure of the chamber and the increasing volatile content therein tends to retard further volatilization while the cellular product sinters at the indicated elevated temperatures. If the furnace is vented during soaking, an insufficient amount of volatile matter may be present to permit inter-particle sintering of the coal particles thus resulting in the formation of a sintered powder as opposed to the desired cellular product.

Additional more conventional blowing agents may be added to the particulate prior to expansion to enhance or otherwise modify the pore-forming operation.

The term "mold", as used herein is meant to define a device for providing controlled dimensional forming of the expanding coal. Thus, any chamber into which the coal particulate is deposited prior to or during heating and which, upon the coal powder attaining the appropriate expansion temperature, contains and shapes the expanding porous coal to some predetermined configuration such as: a flat sheet; a curved sheet; a shaped object; a building block; a rod; tube or any other desired solid shape can be considered a "mold" for purposes of the present invention. Mold materials include glass and ceramics as well as aluminum and steel. As will be explained more completely below, the selection of mold material and consequently heating/volitization rates can affect cell formation and product properties and is consequently an important consideration in the production of the custom designed foams of the present invention.

As will be apparent to the skilled artisan familiar with pressurized gas release reactions, as the pressure in the reaction vessel increases, from 0 psi to 500 psi, as imposed by the non-oxidizing gas, equilibrium will be shifted and the density of the produced coal-based cellular product will increase as the size of the "bubbles" or pores produced in the expanded coal decreases. Similarly, a low soak temperature of, for example about 400°C will result in a smaller pore or bubble size and consequently a more dense expanded coal than would be achieved with a soak temperature of about 600°C. Further, the heat-up rate will also affect pore size, a faster heat-up rate resulting in a larger pore size and consequently a less dense

expanded coal product than a slow heat-up rate. These phenomenon are, of course, due to the kinetics of the volatile release reactions which are affected, as just described, by the ambient pressure and temperature and the rate at which that temperature is achieved as well as the material composition of the mold. These process variables can be used to custom produce the expanded coals of the present invention in a wide variety of controlled densities, strengths etc.

After expanding the coal particulate as just described, the porous coal product is an open celled material. Several techniques have been developed for "sealing" the surface of the open celled structure to improve, for example, its adhesive and permeability characteristics for further fabrication and assembly of a number of parts or for particular utilities. For example, a layer of a commercially available graphitic adhesive can be coated onto the surface and cured at elevated temperature or allowed to cure at room temperature to provide an adherent skin.

After expanding, the porous coal-based preform or product is readily machineable, sawable and otherwise readily fabricated using conventional fabrication techniques.

Subsequent to production of the preform or product as just described, the preform or product may be subjected to carbonization and/or graphitization according to conventional processes to obtain particular properties desirable for specific applications. Activation, for example, by ozone or carbon dioxide, may also be performed, if activation of the coal-based expanded product would be useful in a

final product application such as in filtering of air. Additionally, a variety of additives and structural reinforcers may be added to the coal-based preforms or products either before or after expansion to enhance specific mechanical properties such as fracture strain, fracture toughness and impact resistance. For example, particles, whiskers, fibers, plates, etc. of appropriate carbonaceous or ceramic composition can be incorporated into the porous coal-based preform or product to enhance its mechanical properties.

The open celled, coal-based preforms or products can additionally be impregnated with, for example, petroleum pitch, epoxy resins, phenolic resins or other polymers using a vacuum assisted resin transfer type of process. The incorporation of such additives provides load transfer advantages similar to those demonstrated in carbon composite materials. In effect a 3-D composite is produced that demonstrates enhanced impact resistance and load transfer properties.

The cooling step in the expansion process results in some relatively mimor shrinkage on the order of less than about 5% and generally in the range of from about 2% to about 3%. This shrinkage must be accounted for in the production of near net shape preforms or final products of specific dimensions and is readily determinable through trial and error with the particular coal starting material being used. The shrinkage may be further minimized by the addition of some inert solid material such as coke particles, ceramic particles, ground waste from the coal expansion process etc. as is common practice in ceramic fabrication.

Carbonization is conventionally performed by heating the preform or product under an appropriate inert gas at a heat-up rate of less than about 10°C per minute to a temperature of between about 800°C and about 1200°C and soaking for about 1 hour or less. Appropriate inert gases are those described above that are tolerant of these high temperatures. The carbonization process serves to remove all of the non-carbon elements present in the preform or product such as sulfur, oxygen, hydrogen, etc.

Graphitization, commonly involves heating the preform or product either before or after carbonization at heat-up rate of less than about 20°C per minute, preferably from about 1°C to about 5°C per minute, to a temperature of between about 1700°C and about 3000°C in an atmosphere of helium or argon and soaking for a period of less than about one hour. Again, the inert gas may be supplied at a pressure ranging from about 0 psi up to a few atmospheres.

Through previous work performed in connection with above-referenced U.S. Patent Application Serial No. 09/ 453,729 which is hereby incorporated by reference in its entirety, it has been determined that the cell size of coal-base cellular products is largely determined by the particle size of the starting carbonaceous material. Large cells are produced from large particle size precursors while smaller cells are produced from small particle size precursors. Additionally, it has been discovered that density can be graded or varied by both selection of different precursors or

starting materials as well as thermal gradients within the mold. The latter, as will be shown, below can be controlled by the selection of mold materials to accelerate or decelerate heat up and cool down rates.

Coals suitable for use in the processes described herein are primarily bituminous coals exhibiting a "swell index" as determined by ASTM standards DD5515-97, "Standard Test Method for the Determination of Swelling Properties of Bituminous Coal" and D720-91 "Standard Test Method for Free Swelling Index of Coal" of between about 3 and about 9 and preferably about 4. Best results are achieved in terms of adequate cell generation to obtain coal-based cellular materials of the proper densities when the bituminous coal demonstrates a Gieseler plasticity value commonly characterized as high, i.e. above about 500DDPM. Such values are determined in accordance with ASTM standard D-2639. Agglomerating bituminous coals, i.e. those containing from about 10 to about 32% by weight volatiles are specifically preferred.

In the first proposed application of the processes of the present invention, the overall weight of foams for structural applications can be optimized by locating cellular material of high-density or larger cell wall thickness in regions of high stress, while retaining the lower density or more open-celled structure in less stress-critical regions. This approach can also be used to alter mass properties such as centroid movement, moment of inertia, and radius of gyration of a solid body.

For heat transfer, two methods of design are possible. First, regions of high density or larger cell wall thickness can be included to offer greater heat conduction in desired locations or directions with low density cellular material located to insulate or retard heat flow in other locations or directions. Second, in convective or fluid flow applications, regions of larger, more open celled material can be located to allow gas flow and interaction with graphitic cell walls and ligaments, while higher density or more closed cell material can be used to prevent convective flow in other locations or directions. Such a material could find application in heat exchange applications, for example. These latter materials are also applicable to directed mass transfer applications, for example, in catalysis and electrolyte transfer. In these case, coal-based cellular materials can be designed to allow preferential reactant or electrolyte transfer in a given direction(s) to optimize the efficiency of fuel cells, batteries, catalytic and electrochemical processes. Hence, the coal-based cellular products of the present invention provide the integration of a cellular structure demonstrating a particular strength, heat transfer or mass transfer characteristic in a coal-based cellular matrix of a compositionally similar but characteristically dissimilar coal-based cellular structure.

Coal-based cellular materials of this type, hereinafter called "graded materials" are produced by segregating starting materials having different particle size distributions using partitions placed in the mold during loading. The partitions can then be removed prior to heating without material displacement and the different cell sizes thereby segregated in the chosen regions. Alternatively, the

partitions may comprise materials such as paper that are vaporized during the heating operation, or even carbon meshes or fabrics that become incorporated into the structure during heating.

Density can be similarly graded by two distinct means; 1) use of different precursors and 2) the proper and controlled application of thermal gradients. In the first method, different starting materials are segregated by partitions in the loading operation as just described. For example, part of the mold can be filled with a high volatile bituminous coal and the balance of the mold filled with a low volatile bituminous coal. In this situation, the mass loss and sintering behavior of the two different starting materials can be quite different resulting in graded density within the structure.

In the second proposed application of the processes of the present invention, thermal conditions during cell formation are used to create density gradients within the formed article. For example when cell formation is performed in a thermally insulating mold (glass or ceramic, for example) under rapid heat up conditions, volatiles are largely removed from the top or outer surface areas before the interior material reaches temperature. The surface material thus becomes dense due to being pushed upward or outward by the more slowly expanding internal material. A product formed in such a process will have a dense skin and a less dense core. Thermal conditioning can also be used to produce the opposite effect. If a suitable material is rapidly heated to a very high temperature and/or if the mold is made of a

thermally conductive material such as steel or aluminum, the material adjacent to the mold surface devolatilizes quickly and becomes rigid, i.e. cokes and remains more porous, while the internal structure is being formed and cannot "collapse" the outer surface during expansion as in the case of the slow heat up rate process in an insulating mold just described.

The invention will be better understood when considered in light of the following, non-limiting examples of its implementation.

Examples

Example 1

A high volatile bituminous coal was ground and sieved to produce two distinct particle size ranges; 1) that passing through a 60 mesh screen, and 2) that passing through a 30 mesh screen but not a 35 mesh screen. Equal amounts of both sizes were weighed and poured onto opposite sides of a paper partition that had been placed across the diameter of a circular ceramic mold. After vibrating the mold to achieve proper particle packing, the partition was removewd and the mold loaded into a pressure vessel. The mold under nitrogen pressure of 500 psi was heated to 475°C at a heating rate of 3°C per minute and held at this temperature for seven hours. The resulting cellular product was calcined at 1000°C and then cut to reveal the graded cell structure shown n Figure 1.

Example 2

A high volatile bituminous coal (610 from Pike County, Kentucky) was ground until it passed through an 80 mesh screen, loaded into a steel mold and placed in a pressure vessel containing nitrogen at atmospheric pressure. While this material is known from previous experience to produce a uniform density cellular material when subjected to temperatures between about 425°C and 475°C, it was heated rapidly to 525°C and held at this temperature for seven hours. The resulting structure, showing a low density inner portion and a high density surface is shown in Figure 2.

Example 3

A low volatile bituminous coal was ground until all material passed through a 60-mesh screen. Similarly, a high volatile bituminous coal (610 from Pike County, Kentucky) was ground until it passed through an 80-mesh screen. Equal amounts of both precursors were weighed and poured on opposite sides of a paper partition placed across the diameter of a circular ceramic mold. After vibrating the mold to achieve proper packing, the partition was removed and the mold loaded into a pressure vessel containing nitrogen at 500psi. The mold was heated to 475°C at a slow heating rate (2°C per minute) and held at this temperature for seven hours. The resulting cellular material was cut to reveal the graded cellular structure shown in Figure 3.

Example 4

An NMP (n-methyl pyrollidone) extract of a low volatile bituminous coal was ground until all material passed trough a 60-mesh screen. It was loaded into a ceramic mold and heated slowly to a temperature of 475°C and held for seven hours. The exterior material volatalized first and was pushed outward to the mold surface by the expanding interior giving rise to the pronounced "skin-core" structure shown in Figure 4.

Example 5

A high volatile bituminous coal was ground a sieved to produce tow distinct particle sizes. The first (1) passed through a 60 mesh screen and the second (2) through a 35 mesh but not a 60 mesh screen. A T-shaped mold was constructed from paper and centered in a cylindrical steel mold. The relative areas of the T and the surrounding mold were used to determine the amounts of each material used. In one case, fraction 1 was loaded into the T and fraction 2 into the surrounding mold. In the second case, fraction 2 was loaded into the T and fraction 1 into the surrounding mold. Both were heated to 475°C at a heating rate of 2°C per minute and held at this temperature for seven hours. The resulting cellular materials were cut to reveal the linearly-graded cell structure shown in Figures 5 and 6 respectively.

There have thus been described, coal-based cellular products that can be custom designed to have integral stiffeners or load paths, directed heat transfer paths, and/or directed mass transfer paths and methods for their production. Such

design and production is made possible by the appropriate selection of starting materials, thermal treatment conditions and mold materials combined in at least some instances with segregation of different starting materials in different regions of the forming mold.

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As the invention as been described, it will be apparent to those skilled in the art that the same can be varied in many ways without departing from the sprit and scope of the invention. Any and all such modifications are intended to be included within the scope of the appended claims.

What is claimed is:

1) **A coal-based cellular product comprising a matrix of cells having integral stiffeners or load paths, directed heat transfer paths and/or directed mass transfer paths defined in said matrix by cells of a different density or of a different size.**

2) **The coal-based cellular product of claim 1 prepared from bituminous coal.**

3) **The coal-based cellular product of claim 2 wherein said bituminous coal has a swell index of between about 3 and about 5.**

4) **The coal-based cellular product of claim 2 wherein said bituminous coal has a Gieseler plasticity value above about 500DDPM.**

5) **The coal-based cellular product of claim 1 wherein said stiffeners or load paths, directed heat transfer paths and/or directed mass transfer paths are due to the presence of coal-based cells of a structure differing from those comprising the matrix.**

6) **The coal-based cellular product of claim 1 wherein said stiffeners or load paths, directed heat transfer paths and/or directed mass transfer**

paths are due to the presence of coal-based cells of different densities than those comprising the matrix.

7) The coal-based product of claim 3 wherein said stiffeners or load paths, and mass transfer paths are defined by the presence of coal-based structure of higher density or greater cell wall thickness than that of the surrounding matrix.

8) A method for the production of a coal-based cellular product comprising a matrix of cells having integral stiffeners or load paths, directed heat transfer paths and/or directed mass transfer paths defined in or about said matrix by cells of a different density or of a different size said method comprising:

A) selecting as the matrix material a first coal-based precursor ground to a particle size below about 1mm which matrix material will, upon expansion, provide a matrix of an appropriate strength and density;

B) selecting a second coal-based precursor ground to a particle size below about 1mm, but of a different particle size than that of said matrix, said second coal-based precursor when expanded providing the required integral stiffener or load paths, heat transfer paths and/or mass transfer paths;

C) loading each of said selected coal-based precursors into each of at least two predefined volumes of a mold separated by appropriate partition(s) to define said matrix of said first coal-based precursor having said integral stiffeners or load paths, directed heat transfer paths and/or mass transfer paths defined by said second coal-based precursor in or about said matrix;

D) heating said mold under a non-oxidizing atmosphere to a temperature of between about 300°C and about 700°C and soaking at this temperature for a period of from about 10 minutes to about 12 hours; and

E) controllably cooling said coal-based product.

9) The method of claim 8 wherein said partitions are removed prior to initiation of said heating.

10) The method of claim 8 wherein said partitions remain in place during said heating and are either integrated into the coal-based product or vaporized.

11) The method of claim 8 wherein said mold comprises glass or ceramic.

12) A method for the production of a coal-based cellular product comprising a matrix of cells having integral stiffeners or load paths,

directed heat transfer paths and/or directed mass transfer paths defined by or about said matrix by cells of a different density comprising:

A) placing a coal-based precursor ground to a particle size below about 1mm into a thermally conductive mold;

B) placing said mold into a pressure chamber under a non-oxidizing atmosphere;

C) heating said mold at a rapid heat-up rate to a temperature of between about 300°C and about 700°C and soaking at this temperature for a period of from about 10 minutes to about 12 hours; and

D) controllably cooling said coal-based product

to provide a product comprising a relatively less dense coal-based cellular core surrounded by a relatively more dense and therefore less permeable skin.

13) The method of claim 12 wherein said mold comprises aluminum or steel.

Abstract

Coal-based cellular products that can be custom designed to have integral stiffeners or load paths, directed heat transfer paths, and/or directed mass transfer paths and methods for their production are described. Such design and production is made possible by the appropriate selection of starting materials, thermal treatment conditions and mold materials combined in at least some instances with segregation of different starting materials in different regions of a forming mold.

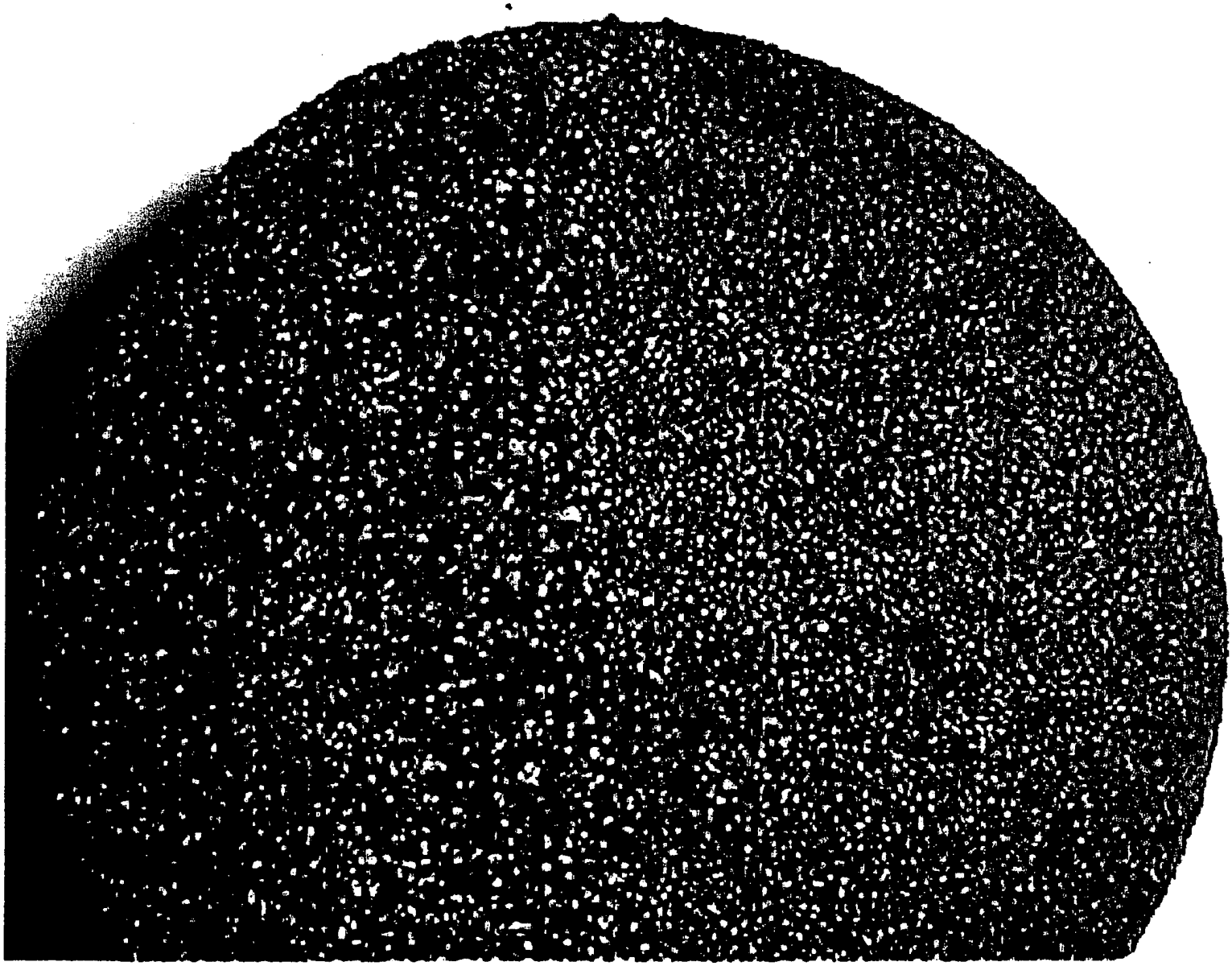

FIGURE 1

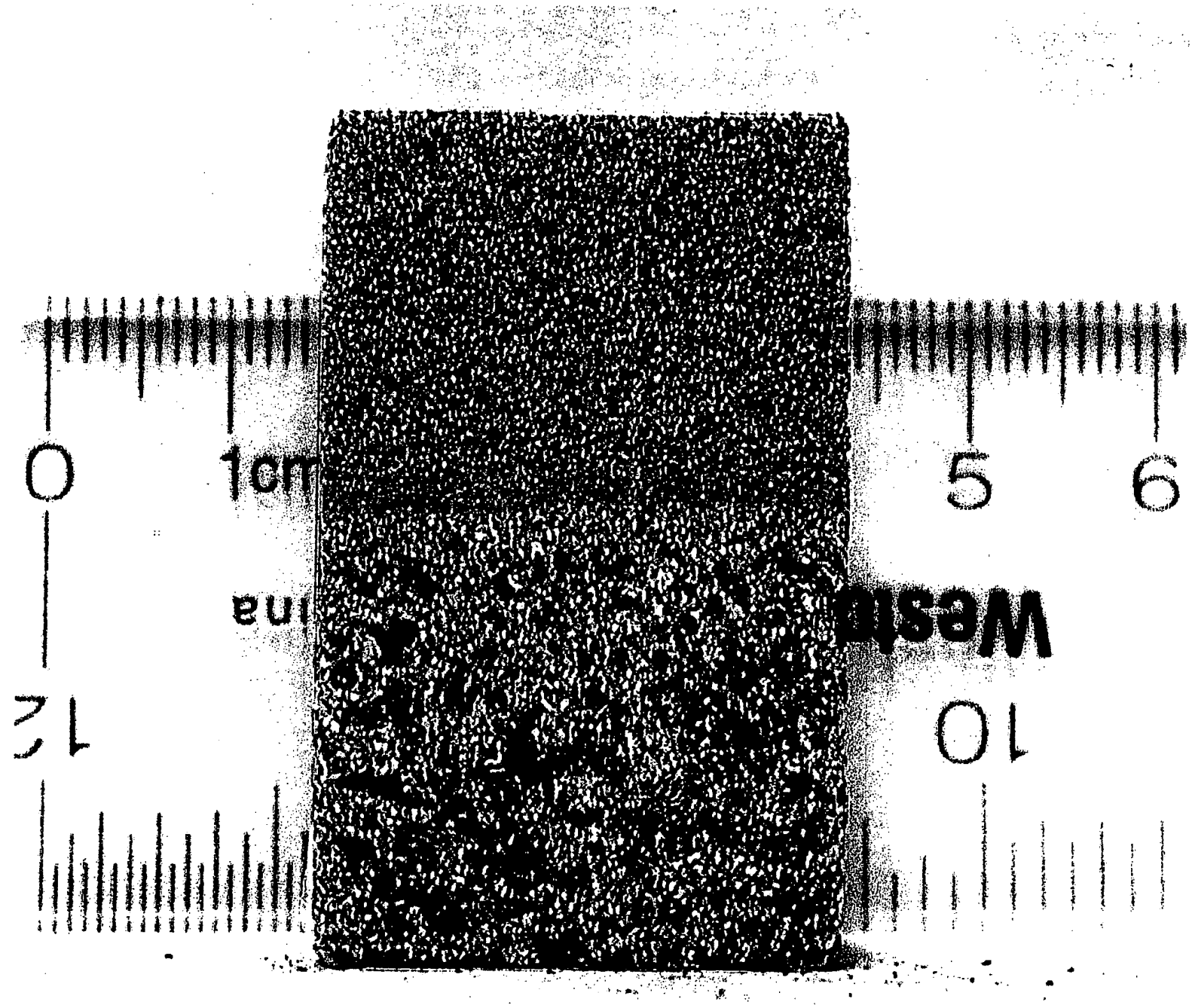

FIGURE 2

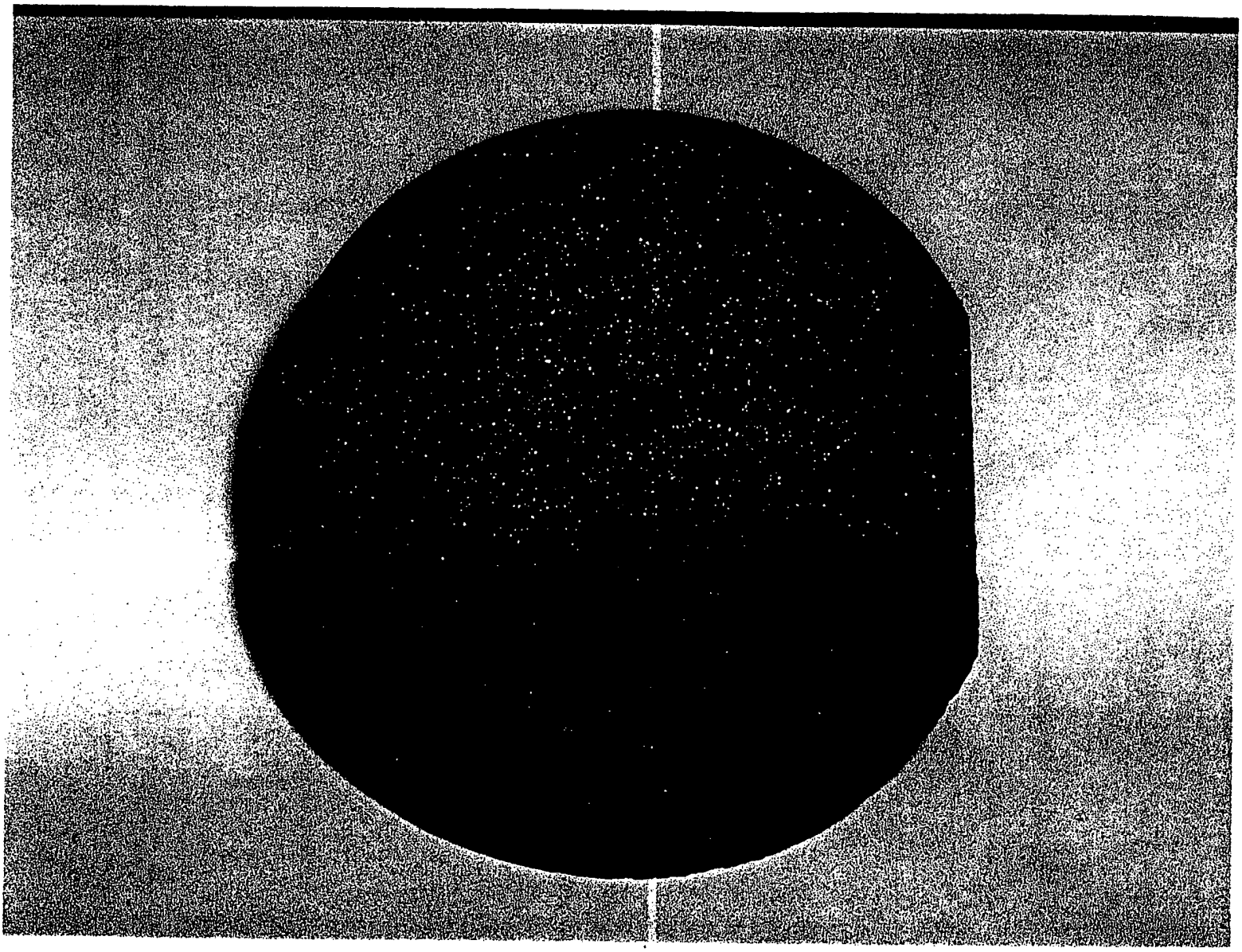

Figure 3

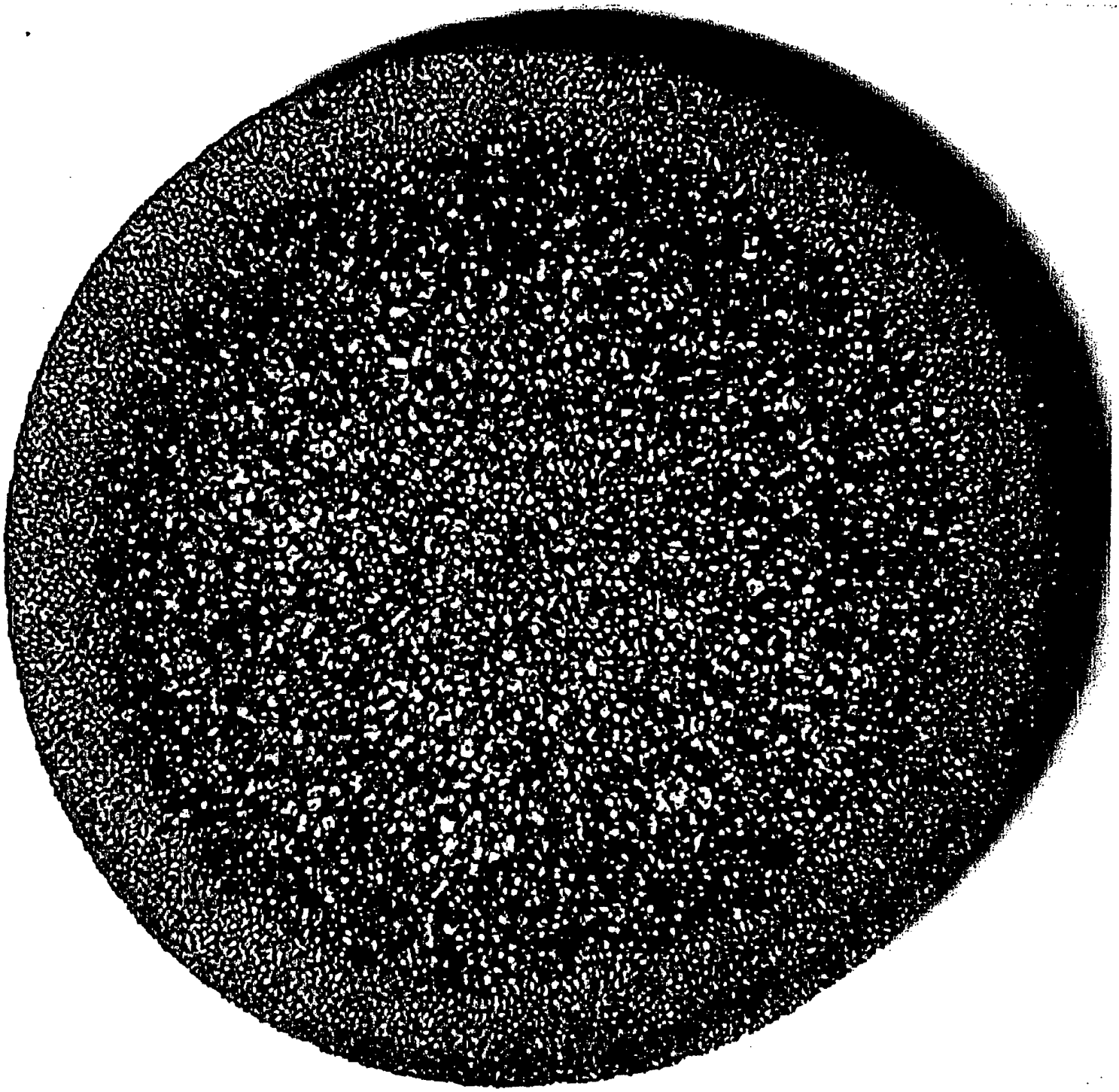

Figure 4

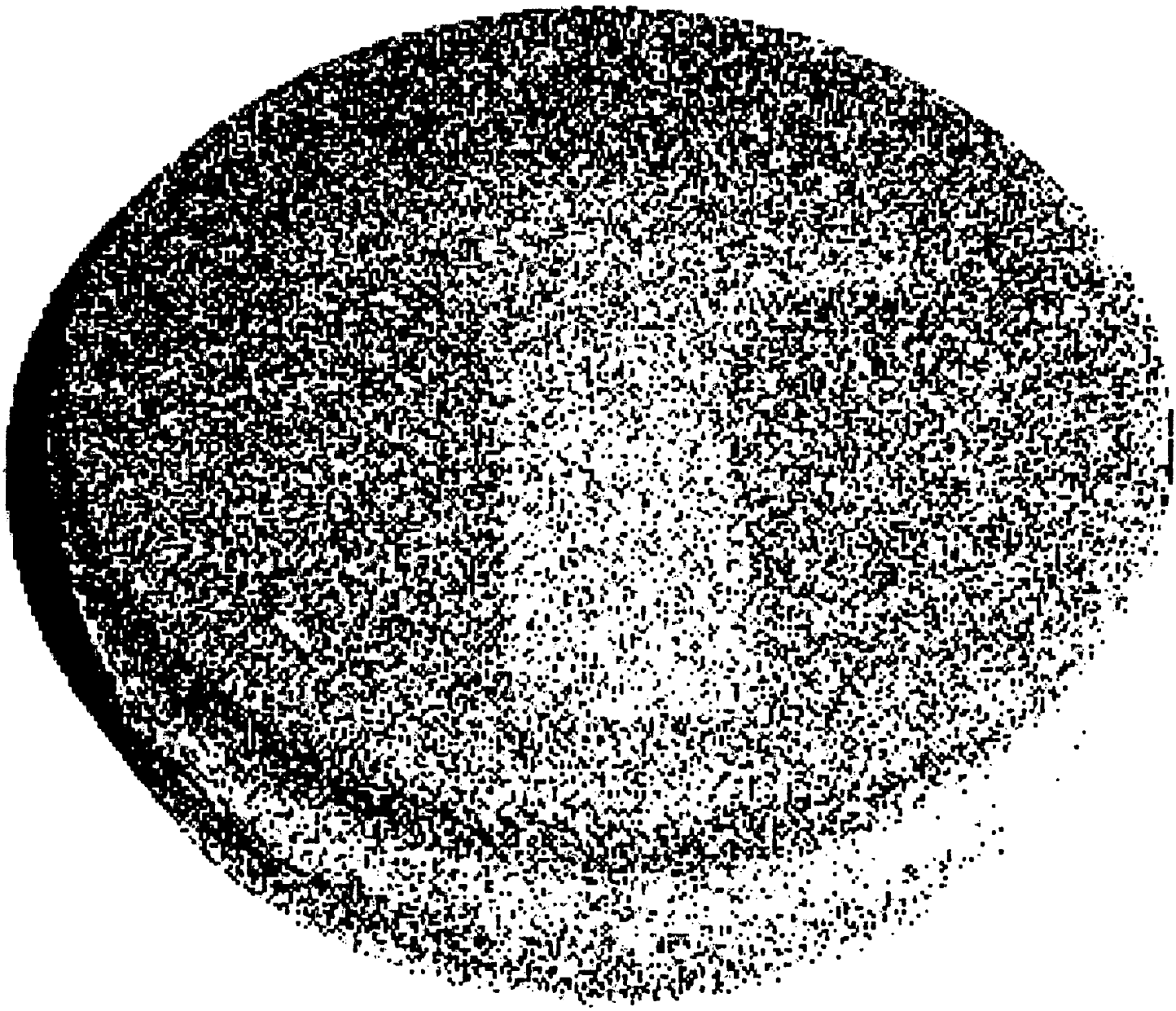

Figure 5

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APPLICATION NUMBER	FILING DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	DRAWINGS	TOT CLAIMS	IND CLAIMS
09/733,602	12/08/2000	1714	420	1391 (Touchstone)	5	13	3

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Date Mailed: 05/16/2001

Receipt is acknowledged of this nonprovisional Patent Application. It will be considered in its order and you will be notified as to the results of the examination. Be sure to provide the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION when inquiring about this application. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please write to the Office of Initial Patent Examination's Customer Service Center. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections (if appropriate).**

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**** SMALL ENTITY ****

Title

Designed cellular coal

Preliminary Class

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NOTICE OF INFORMAL APPLICATION

This application is considered to be informal since it does not comply with the regulations for the reason(s) indicated below. The period within to correct the informalities noted below and avoid abandonment is set in the accompanying Office action.

A new oath or declaration, identifying this application number is required. The oath or declaration does not comply with 37 CFR 1.63 in that it:

- does not identify the residence (e.g., city and either state or foreign country) of each inventor.

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Exhibit B